

PROJECT TITLE : QA ANALYTICAL SERVICES
PERIOD COVERED : MAY 21 - JULY 21 1981
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1.1. METHODS

- Determination of the moisture content of tobacco. (1)
The moisture content of different samples was determined by PME method No. 210 (oven volatiles) and the "Coresta" reference method (azeotropic distillation with cyclohexane). The following results were obtained:

Sample	Distil- lation, %	O.V. (%)	Δ Dist.-O.V. (%abs.)	Δ Dist.-O.V. (% rel.)
ETNA-FC (after tower)	2.9	4.4	+ 1.5	+ 52
ETNA-FC (final product)	8.8	9.9	+ 1.1	+ 13
ETNA-FC (cut rag)	18.8	19.8	+ 1.0	+ 5.3
MLF-blend (before flavouring)	9.7	11.4	+ 1.7	+ 18
MLF-blend (after flavouring)	9.7	11.2	+ 1.5	+ 16
MAA-blend (before flavouring)	9.1	10.0	+ 0.9	+ 10
MAA-blend (after flavouring)	9.1	10.2	+ 1.1	+ 12
BRD-blend	8.7	9.3	+ 0.6	+ 6.9

The standard deviations for the two methods are similar. The results led to the following conclusions:

- The values obtained by distillation are in any case lower than the O.V. - values.
- The higher the quantity of ingredients in a blend, the higher is the difference (in relative %) between the two methods (comparison between MLF-, MAA- and BRD-blends).
- The lower the moisture content of a sample, the higher is the difference between the two methods and vice-versa. This means that in the O.V.-method, samples with a low moisture content are "over-dried" and samples with a high moisture content are not "sufficiently dried".

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1.4. ORGANISATION

- Control of ESTHER-production

Processing control of ESTHER-production has been carried out since June 1, 1981, by the QA-Laboratory in Onnens instead of by the QC-Preparation Laboratory in Serrières.

2.1. CASING KITCHEN

- BSE-AC (2)

The preparation procedure for the new solution of BSE-AC was established. Because of its strong odour, the solution must be prepared in a specially indicated tank reserved for this purpose.

- Determination of Density and Refraction in solutions (3).

The purpose of this work was to get an idea as to what extent and reliability solutions may be controlled by the determination of density and refraction. The study was made on MF-PC solutions produced in the laboratory.

Individually, each component was applied at different rates (0 %, 50 %, 80 %, 90 %, 110 %, 120 % and 150 % of the normal quantity). The final volumes of each solution (according to the standard recipe) were kept identical by completing them with water. Afterwards the density and the refraction were measured. In graphs the results were plotted against the quantity of the individual components. By introducing the standard deviation for MF-PC, obtained during the control trial (see below), the following detection limits were found:

Ingredient	in % of the standard quantity	
	<u>by refraction</u>	<u>by density</u>
Glycerine	95 - 103	97 - 105
Propylene glycol	93 - 103	70 - 130
Honey	91 - 113	93 - 105
Invert sugar	88 - 110	95 - 105
Water(final volume)	91 - 105	90 - 108

In a second step, where the same principles as those described above were applied, inversions between two similar components were examined.

Ingredient	theore-tical rate(%)	detection limits as % of the theoretical rate	
		by refraction	by density
Glycerine	55	38 - 72	53 - 58
Propylene glycol	45	28 - 62	42 - 47
Honey	48	16 - 79	26 - 69
Invert sugar	52	21 - 84	31 - 74

In conclusion, even though with other solutions the composition and the interferences may be different, we can say that:

- Important errors in the quantities of the main components can be detected by combining the two methods.
- Errors in the addition of low quantity components cannot be detected (eg artificial honey flavour).
- Inversion of similar components can easily be detected in the case of humectants, but not in the case of honey and invert sugar.

- Quality Control of solutions (4)

Density and refraction values were determined systematically in all the solutions produced in the casing kitchen during two trials carried out over a period of about 3 months. The statistical evaluation of the results led to the following conclusions:

- Both shifts work identically and follow the fabrication procedures.
- Both shifts are at the same level as regards quality of solutions produced.

On the basis of the results obtained, specifications for the most important solutions (BURLEY CASING, BURLEY TOP FLAVOUR, MF-PC, etc.) were established. Furthermore, measures to improve quality, to eliminate potential danger of errors and to facilitate manipulation and working facilities were proposed and discussed with production staff (5).

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3.2.1. TRIALS WITH NEW SUPPLIES OF TOBACCO INGREDIENTS

- Invert sugar (SCHOLTEN/AVEBE, Foxhol(NL); "TRICOLIN Z 3833" / PMH Bergen op Zoom) (6)

The sample is within specifications.

- Licorice extract (MAC ANDREWS & FORBES, Croydon (USA); produced in IRAN/yellow sheet 5923, 50 kg). First industrial trial (MLF-CH)

Laboratory: The sample is slightly too high as regards the content of glycyrrhizic acid.

Smoke analyses: No significant difference between the standard and the trial was found.

Subjective evaluation: A significant difference between the standard and the trial was found. The trial was rejected.

Concluded on June 15, 1981

- Licorice extract (FERTILIZER & CHEMICALS, Haifa (Israel)/ yellow sheet 6000, 50 kg) First industrial trial (MLF-CH)

Laboratory: The sample is within the specifications.

Smoke analyses: No significant difference between the standard and the trial was found.

Subjective evaluation: A significant difference between the standard and the trial was found. The trial was rejected.

Concluded on June 15, 1981

- Licorice extract (PAULIN PAUL Ltd., London; produced in CHINA (TIENTSIN)) (7)

The content of ash is too high. In addition to this, a significant difference in taste and odour, compared with the standard, was detected.

- Raw cane sugar (A. TOEPFER & CO, Hamburg; produced on FIDJI-ISLANDS/yellow sheet 6030, 30 kg) First industrial trial (MLF-CH)

Laboratory: The sample is within the specifications.

Smoke analyses: No significant difference between the standard and the trial was found.

Subjective evaluation: A significant difference between the standard and the trial was found. The trial was rejected.

Concluded on June 15, 1981

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- Raw cane sugar (A. TOEPFER & CO, Hamburg; produced in JAMAICA) (8)

The sample is within the specifications.

- Raw cane sugar (A. TOEPFER & CO, Hamburg; produced in TRINIDAD) (8)

The sample is within the specifications.

- Sodium benzoate (C.D.f. CHIMIE, Paris) (9)

The sample is slightly out of specifications as regards the water content.

- Tobacco Flavour 15.96.0242 (IFF AG, Reinach (CH)/yellow sheet 6078, 120 kg)

The product was accepted as regards subjective evaluation (Panel A) of the first production of BSE and BSH.

3.2.2. QUALITY CONTROL OF TOBACCO INGREDIENTS

- Ingredients used in LECCE for DIANA-production (10)

In a trial in February 1981 it was found that the cocoa powder and the licorice in blocks were out of specifications. Therefore samples of the two products of the last shipment were submitted for analyses:

- Licorice in blocks is slightly out of specifications as regards water and glycyrrhizic acid, but is nevertheless acceptable.

- Cocoa powder is identical with the first sample, this means largely out of specifications as regards the fat content (10 % instead of 20 - 26 %).

- Ingredients used in PMG Berlin (11)

Because of taste problems with the MLK-DB test cigarettes, the ingredients used were submitted to an analytical checking.

All the products were within specifications (invert sugar, glycerine, propylene glycol, chocolate liquor, ethanol) or were comparable with preliminary shipments (MFSBC Powder, Honey Flavour). Certain doubts about MFSB 1 - Liquid, as regards odour and GC-fingerprints, were eliminated after a local panel test in Munich.

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- SFC 152 (PMG Munich) (12)

Concerning taste problems on PMS-cigarettes produced in February 1981, it was found that the base used for AC-solution easily separates into layers.

General information was given to users to mix well the contents of the drums containing the bases before use (13).

3.4.1. TRIALS WITH NEW SUPPLIES OF FILTER COMPONENTS

- Activated Charcoal (DEGUSSA(Germany), "DESOREX") (14)

The sample is largely out of specifications as regards CCl₄ - index.

- Polyethylene glycol 600 (EIGENMANN & VERONELLI, Milano; produced by CHEMISCHE WERKE HÜLS, Marl (Germany)) (14)

The molecular weight is within the specifications. However, the acidity and water content are slightly out of the specifications.

Another sample for a second analysis will be ordered directly from the producer (15).

3.4.2. QUALITY CONTROL OF FILTER COMPONENTS

- Triacetin for filter production of MLF-cigarettes in Bulgaria (16)

The sample corresponds to our specifications.

3.5.1. TRIALS WITH NEW SUPPLIES OF FILTER GLUES

- Liquid glue for KDF III (filter rod seam) (HENKEL & CIE AG, Pratteln (CH); "7693" and "7630"/ yellow sheet 6057, 2 x 10 kg)

Machineability: The type "7630" was only applicable at reduced machine speed. After addition of 3 % water to the type "7693", filter plugs were produced at the maximum speed.

Subjective evaluation (only for the glue "7693"): A significant difference between the trial and the standard was found. The trial was rejected.

A further trial with another type from this producer is planned.

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3.8.1. TRIALS WITH NEW SUPPLIES OF PACKING GLUES

- A MOLINS HL packing machine in Cousset was transformed to a higher speed (250 HLP/min). Because of gluing difficulties with the standard glue, a series of trials with alternative products was organized. When the trials were run the machine speed was at 235 HLP/min.

- "PLUS RAPIDE" (W. LAESSER, Erlinsbach/yellow sheet 6017, 25 kg)

Machineability: The glue is not better than the standard.

- "V 6" (W. LAESSER, Erlinsbach/yellow sheet 6027, 25 kg)

Machineability: The glue is not better than the standard.

- "V 3" (W. LAESSER, Erlinsbach/yellow sheet 6027, 25 kg)

Machineability: A significant improvement in comparison with the standard glue was found.

300 kg of this glue were ordered for a long-term test (May 11, 1981).

- Glue for the flap of the "export" case (W. LAESSER, Erlinsbach; "LESSO 1138" and "LESSO 1138-67" / yellow sheet 5974, 2 x 20 kg)

The glue is applied semi-automatically with a spray gun.

"LESSO 1138-67" showed better properties than "LESSO 1138".

300 kg of the first were ordered for a long-term test.

- Glue for the flap of the "export" case (W. LAESSER, Erlinsbach; "LESSO 1138-67" / yellow sheet 5981, 300 kg)

The first results were confirmed. The glue has been accepted as a standard glue.

4.1.1. TRIALS ON PRE-CUTTING SOLUTIONS

- Artificial honey in MF-PC solution

An industrial trial on MLF-cigarettes with 100 % artificial honey was organized.

Smoke analyses: No significant difference between the standard and the trial was found.

Subjective evaluation: A significant difference between the trial and the standard was found. The trial was rejected. All the test cigarettes with 100 % artificial honey seemed to have the same defect: They "dried" the mouth.

Based on the result of the subjective evaluation, trials with different amounts of honey flavour on the basis of 100% artificial honey are planned.

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5.1. PRODUCT QUALITY

- Spots on MLF-cigarettes (17)

In a normal production, spots on MLF-cigarettes were observed.

It was found that neither the humectant content nor the moisture content was irregular. Also the applied reconstituted sheets (RCB US and CH) were comparable with preliminary shipments.

As the spots were UV-active, they had been caused by a machine oil. By means of UV-spectra, the product was identified as an oil which is used on cigarette machines (MACOMA 150).

5.3. ASSISTANCE TO OTHER COUNTRIES

- Analyses for the ETNA-plant PMG Munich
Humectants in tobacco (18 samples)
- Analyses for the ETNA-plant PMH Bergen op Zoom
Humectants in tobacco (30 samples)

5.4. SERVICES FOR OTHER GROUPS

- Analyses for Process Development
 SiO_2 in dust and tobacco (19 samples)
Chlorides in tobacco (11 samples)
Ash in tobacco (11 samples)
Calcium, Potassium and Magnesium in tobacco (11 samples)
- Analyses for Product Development
Plasticizer on filters by GC (13 samples)
- Analyses for Research Department
HWS in tobacco (6 samples)
Chlorides in solutions (45 samples)

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7. Letter from Widmer-A to Froideveaux-R (June 12 1981)
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11. Letter from Widmer-A to Tessendorf-W (May 13 1981)
12. Letter from Schwarb-A to Lopes-F (March 24 1981)
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13. Letter from Lopes-F (March 17 1981)
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